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Novel Microfiber Sensor and Its Biosensing Application for Detection of hCG Based on a Singlemode-Tapered Hollow Core-Singlemode Fiber Structure

Ling Chen, Bin Liu*, Juan Liu, Sheng-Peng Wan, Tao Wu, Jinhui Yuan*, Xian Zhou, Keping Long, Liyang Shao, Yong Qing Fu, Xing-Dao He and Qiang Wu*

Abstract— A novel microfiber sensor is proposed and demonstrated based on a singlemode-tapered hollow core-singlemode (STHS) fiber structure. Experimentally a STHS with taper waist diameter of 26.5 μm has been fabricated and RI sensitivity of 816, 1601.86, and 4775.5 nm/RIU has been achieved with RI ranges from 1.3335 to 1.3395, from 1.369 to 1.378, and from 1.409 to 1.4175 respectively, which agrees very well with simulated RI sensitivity of 885, 1517, and 4540 nm/RIU at RI ranges from 1.3335 to 1.337, from 1.37 to 1.374, and from 1.41 to 1.414. The taper waist diameter has impact on both temperature and strain sensitivity of the sensor structure: (1) the smaller the waist diameter, the higher the temperature sensitivity, and experimentally 26.82 $\mu\text{m}/^\circ\text{C}$ has been achieved with a taper waist diameter of 21.4 μm ; (2) as waist diameter decrease, strain sensitivity increase and 7.62 $\text{pm}/\mu\text{e}$ has been achieved with a taper diameter of 20.3 μm . The developed sensor was then functionalized for human chorionic gonadotropin (hCG) detection as an example for biosensing application. Experimentally for hCG concentration of 5 mIU/ml, the sensor has 0.5 nm wavelength shift, equivalent to limit of detection (LOD) of 0.6 mIU/ml by defining 3 times of the wavelength variation (0.06 nm) as measurement limit. The biosensor demonstrated relatively good reproducibility and specificity, which has potential for real medical diagnostics and other applications.

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Ling Chen, Bin Liu, Juan Liu, Sheng-Peng Wan, Tao Wu and Xing-Dao He are with the Key Laboratory of Nondestructive Test (Ministry of Education), Nanchang Hangkong University, Nanchang 330063, China. (e-mail: liubin@nchu.edu.cn).

Jinhui Yuan, Xian Zhou and Keping Long are with the Research Center for Convergence Networks and Ubiquitous Services, University of Science & Technology Beijing, Beijing 100083, China. (e-mail: yuanjinhui81@bupt.edu.cn).

Liyang Shao is with Department of Electrical and Electronic Engineering, Southern University of Science and Technology of China, Shenzhen, China

Qiang Wu and Yong Qing Fu are with the Faculty of Engineering and Environment, Northumbria University, Newcastle upon Tyne, U.K. Qiang Wu is also with the Key Laboratory of Nondestructive Test (Ministry of Education) of Nanchang Hangkong University, Nanchang 330063 (e-mail: qiang.wu@northumbria.ac.uk).

Index Terms— Hollow core fiber, refractive index, strain, temperature, biosensor.

I. INTRODUCTION

OPTICAL fiber sensors are widely applied in various aspects such as physical, biomolecule, chemical and food-borne pathogenic bacteria detection because of their advantages such as high sensitivity, immunity to electromagnetic interference, and ease of fabrication [1-3]. There are different optical fiber sensor structures such as singlemode-multimode-singlemode (SMS) fiber structure [4], surface plasmon resonance (SPR) [5-7], fiber Bragg gratings (FBG) [8-11], long-period gratings (LPG) [12, 13] and optical fiber coupler [14].

Hollow core fiber (HCF) is a simple cylindrical light guiding structure consisting of a hollow air part in the center and a quartz tube wall, which has attracted extensive attention and research interest due to its unique property of air core compared with traditional solid core fiber [15]. The principles of HCF based sensor are mainly divided into three types, namely anti-resonance, Fabry-Perot and multimode interference [16-17]. For example, Liu *et al* proposed a high-temperature sensor based on the combination of single mode fiber (SMF) and HCF, namely SMF-HCF-SMF anti-resonance structure which also can be employed in sub-micrometer resolution liquid level [18-19]. Zhang *et al* utilized SMF- short HCF-SMF to form extrinsic Fabry-Perot interferometer for strain sensing [20]. Duan *et al* proposed in-fiber Fabry-Perot and Mach-Zehnder interferometers based on HCF fabricated by arc fusion splicing with small lateral offsets for temperature measurement, but the fatal drawback of this structure is fragile and poor reproducibility because of center offset splicing [21]. Zhang *et al* compared three RI interferometers with different inner diameters (5 μm , 15 μm , 25 μm) based on SMF-HCF-SMF interference structure [22], which has relatively low RI sensitivity.

Human chorionic gonadotropin (hCG) is a multi-functional molecule that plays an important role in pregnancy, fetal growth, pituitary secretion and cancer cell biology [23]. It has been reported that hCG also plays a role in promoting uterine angiogenesis during pregnancy in order to ensure maximum blood supply to the invading placenta which is an important

function during pregnancy [24]. It is a cancer promoter in all human malignancies and is a supplement to pituitary LH during the maternal menstrual cycle [25]. In 2019, kumar et al reported a novel hCG detection technology based on magnetic microspheres enhanced microfiber interferometer [26]. Therefore, it is extremely necessary to accurately detect the concentration of hCG in the human body.

In this paper, we proposed to taper the HCF fiber section to improve RI sensitivity. Theoretical study of the mode interferometer properties and spectral responses of RI are provided by using the beam propagation method (BPM). Furthermore, the influence of waist diameter on temperature and strain responses is experimentally investigated. Finally, highly sensitive and specific detections of hCG are analyzed in detail based on the proposed fiber structure.

II. THEORETICAL ANALYSIS

Figure 1 shows a schematic diagram of the STHS fiber structure. A short section of HCF is fusion spliced between two traditional SMFs and the center of the HCF section is tapered to a small diameter. The HCF has a core diameter of 10 μm , which is very close to that of the SMF (9.2 μm). The fusion splice will introduce slightly air core collapse when connecting the HCF to SMF. As light injected from the input SMF to HCF, part of light will be transmitted as guided mode within cladding of the tapered HCF (between the air core and surrounding material). These modes will interfere each other, resulting in wavelength shift of interference dip once the surrounding RI changes. The rest light will be transmitted into the air core of HCF, which will act as leaky mode and disappear after transmitting to the output SMF. Hence, for the STHS fiber structure, the principle of transmission is the guided cladding modes within tapered HCF section, where the silica cladding of the tapered HCF acts as a core of the waveguide and both the air core and surroundings act as cladding of the waveguide.

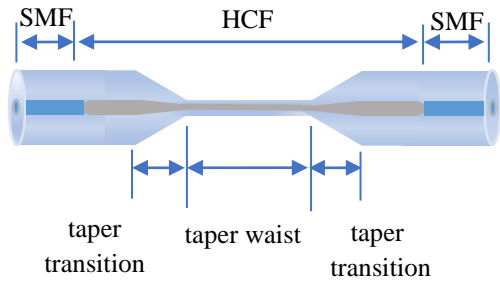
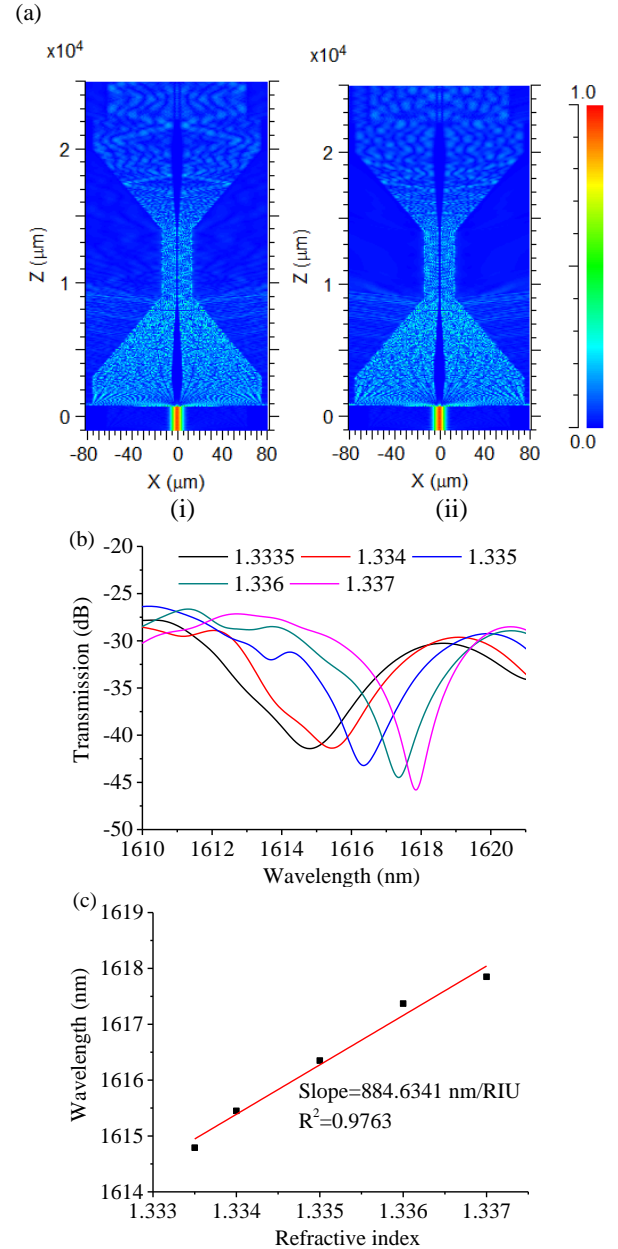


Fig. 1. A schematic diagram of the proposed STHS fiber structure

Numerical simulations based on the STHS fiber structure was carried out by using BPM. The simulation conditions are based on the mesh size of the 2D model in the X and Z directions, the grid size is set to 0.1 and 1 μm , respectively. The model boundary conditions are based on the perfectly matched layer condition. The core and cladding diameters of the SMF is set to 9 and 125 μm , and the corresponding RI are 1.4507 and 1.4428, respectively. The HCF has RI of 1.0 and 1.4428, and diameter of 10 μm and 150 μm , for core and cladding respectively. The lengths of taper transition and taper

waist section of the HCF (21 mm) are set to 6 and 5 mm, respectively. The taper waist diameter is 26.5 μm . The air hole become collapsed from 10 μm to 5 μm at the interface between SMF and HCF. Figure 2(a) (i) and (ii) show the distribution of the optical field propagating along the STHS fiber structure when free space wavelengths are 1614.79 nm (dip) and 1618.63 nm (peak) at the surrounding RI of 1.3335, which indicate an obviously mode interference within the tapered HCF section. As light incident from SMF to air hole collapsed HCF, most light coupled into the cladding layer of HCF as guided mode and transmitted within the tapered HCF section. However, at the interface between HCF and output SMF, very limited light coupled back to the SMF. Figures 2(b), (c) and (f) show that the simulated spectra red shift the as the external RI increases at three different external RI ranges from 1.3335 to 1.337, from 1.37 to 1.374, and from 1.41 to 1.414. The RI sensitivities are calculated as 885, 1517, and 4540 nm/RIU [shown in Figs. 2(c), (e), and (g)], respectively.



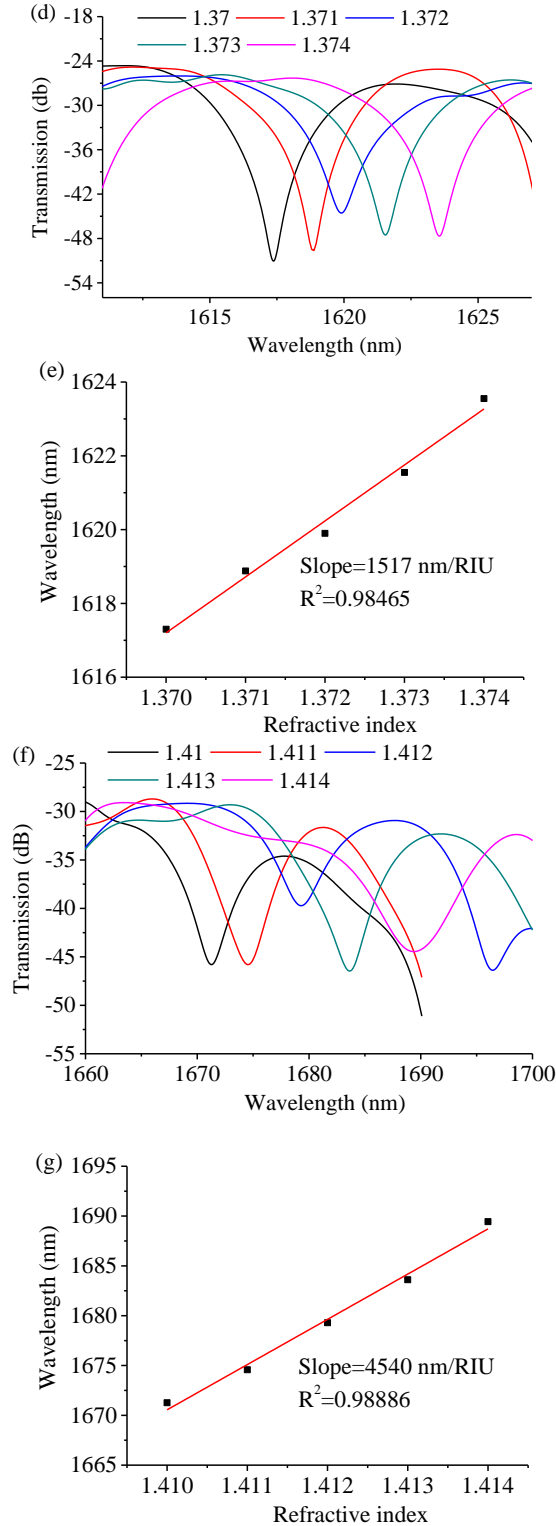


Fig. 2. Simulated results of the STHS fiber structure with taper waist diameter of 26.5 μm : (a) Optical field; (b) spectral response at external RI range of: (b) 1.33; (d) 1.37; (f) 1.41; and calculated RI sensitivity at RI range: (c) 1.33; (e) 1.37; (g) 1.41.

III. EXPERIMENTAL INVESTIGATION

The STHS Fiber Structure for RI Sensing

A length of 10 mm HCF with 10 μm air core diameter and 150 μm cladding diameter is spliced with two SMFs by using a

commercial fusion splicer (Fujikura 80C). The manual splice mode is used to combine the HCF with SMF, and parameters of the manual fusion are listed in Table 1. Due to the surface tension, part of air hole near fusion splice points between the SMF and HCF would collapse. Then, the SMF-HCF-SMF structure is tapered to small diameter by a commercial optical fiber tapering system (OC-2010, JILONG).

TABLE 1
FUSION SPLICE PARAMETERS USED IN THE EXPERIMENT

Discharge time	Overlapping	Discharge power
100 ms	8 μm	standard +8 bit

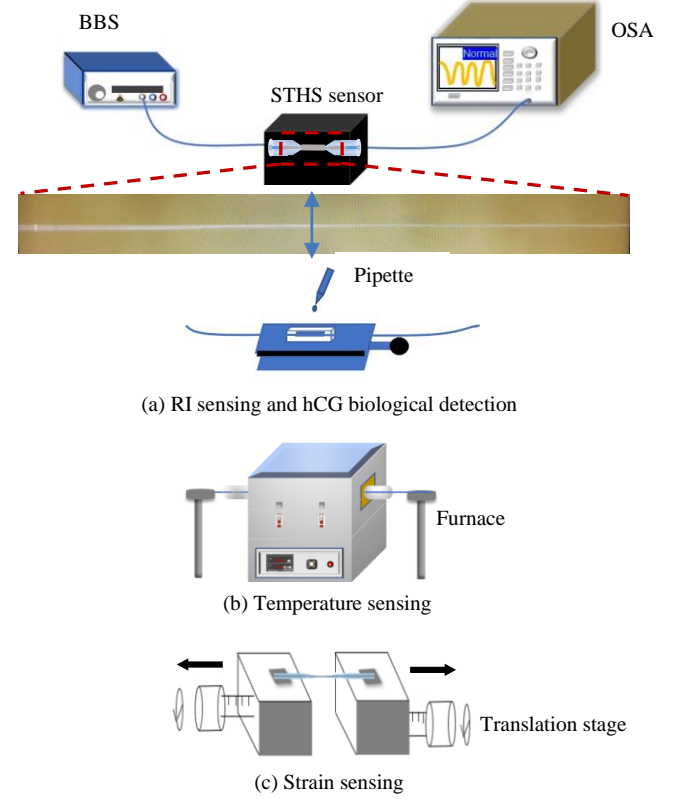


Fig. 3. The schematic diagram of the experimental setup (a) RI sensing and hCG detection, (b) temperature measurement and (c) strain sensing

Firstly, the influence of the taper waist diameter on RI response was investigated with three different tapered waist diameters of 150 (untapered), 59.3 and 26.5 μm . A schematic diagram of the experimental setup is shown in Fig. 3. The light supplied by a broadband source (BBS, SC-5-FC) is transmitted through the STHS fiber sensor and detected by an optical spectrum analyzer (OSA, YOKOGAWA AQ6370D). Figure 4(a) show the spectral responses of the STHS fiber structure with a taper diameter of 26.5 μm vs. RI at range of 1.33. The comparison of RI sensitivities with different tapered waist diameters is shown in Fig. 4(b). With increase of surrounding RI, wavelengths of dips are monotonically redshift for all the three tapered waist diameters, and the RI sensitivities increase from 128 nm/RIU to 816 nm/RIU as the taper diameters decrease from 150 to 25.6 μm . We also test RI

responses at RI ranges from 1.369 to 1.378 and from 1.409 to 1.4175 with a taper waist diameter of 25.6 μm in Figs. 4(c) and (d), RI sensitivities of 1601.86 and 4775.5 nm/RIU were achieved, which agree very well with numerical simulation results [Figs. 2(e) and (g)]. In addition, a stability test has been carried out by immersing the fabricated STHS fiber sensor into phosphate buffered saline (PBS) buffer [shown in Fig. 4(e)]. The results indicate that the STHS sensor has very good stability with wavelength variations of ± 0.02 nm over 35 minutes.

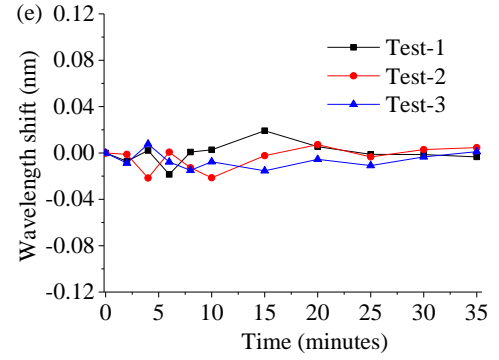
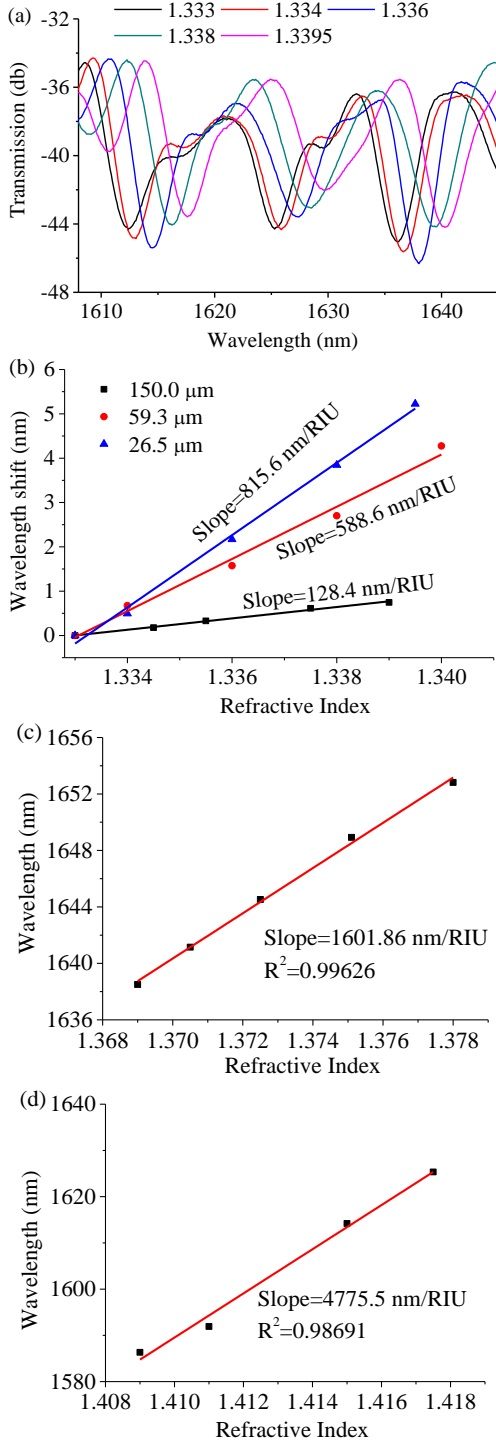
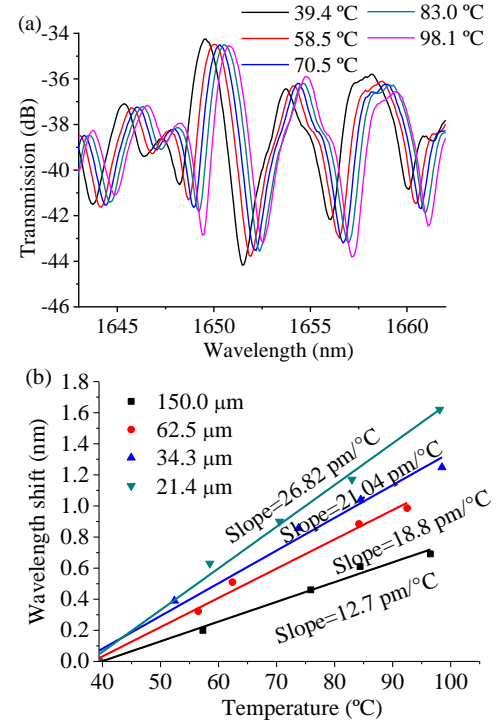


Fig. 4. Measured (a) spectral response of the sensor vs. RI variations at RI range of 1.33; wavelength shift of the sensor at RI range of (b) 1.33 (c) 1.37; (d) 1.41; and (e) stability tests of the sensor in PBS buffer.

The STHS fiber structure for temperature and strain sensing

In the following, temperature and strain sensing performances of the STHS fiber structure were tested experimentally. The experimental device diagram for temperature and strain sensing are shown in Figs. 3(b) and (c). Figure 5(a) shows the spectral responses with taper waist diameter of 21.4 μm at different temperatures, where wavelength shifts towards longer wavelengths monotonically as temperature increases from 39 to 98 $^{\circ}\text{C}$. The influence of taper waist diameter on temperature response is shown in Fig. 5(b). As the waist diameters decrease from 150 to 21.4 μm , the temperature sensitivities gradually increase from 12.7 to 26.82 $\text{pm}/^{\circ}\text{C}$. Then, the spectral responses when different strain was applied to the sensor are shown Fig. 5(c). The wavelength undergoes blue shift with growth of strain. Figure 5(d) shows the influence of the taper waist diameter on strain response. As the diameters decrease from 150 to 20.3 μm , the strain sensitivities increase from 1.34 to 7.62 $\text{pm}/\mu\epsilon$.



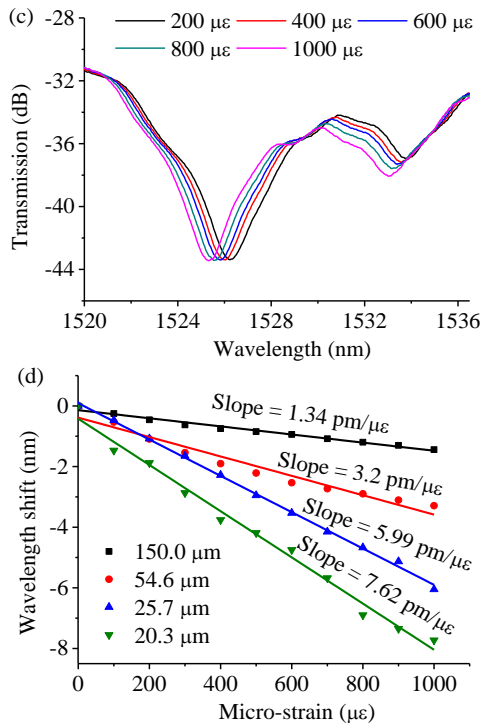


Fig. 5. Measured (a) spectral responses at different temperatures; (b) wavelength shifts vs. temperature with different taper waist diameter; (c) spectral responses at different strain; (d) wavelength shifts vs. strain with different taper waist diameter.

Functionalization of the STHS fiber structure for hCG detection

As studied above, the STHS fiber sensor with a diameter of 26.5 μm has maximum RI sensitivity, hence was selected for biosensing application. Before functionalizing the fiber sensor, the STHS fiber sensor was immersed into potassium hydroxide (KOH)-ethanol standard solution to clean the surface of the fiber for one hour at room temperature. It was then washed several times with deionized water and compared with the pH indicator paper until it was neutral (pH=7.0). The fiber sensor was then functionalized following the four steps below:

- Immerse the fiber sensor in 5% 3-(3-triethoxysilylpropyl) oxolane-2,5-dione (silanization reagent) for four hours to produce carboxyl group on the fiber surface.
- Wash the fiber sensor with pH buffer standards DIN 19266 values (PB, pH=6.0), then immerse into the mixed solution of 1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (0.8 mg/ml EDC) and hydroxy-2,5-dioxypyrolidine-3-sulfonic acid sodium salt (1.2 mg/ml NHSS) for one hour to obtain NHS active ester.
- The above treated fiber sensor is then immediately immersed into hCG- β -mAb solution with concentration of 15 $\mu\text{g}/\text{ml}$ for four hours to immobilize the hCG- β -mAb on the fiber sensor surface.
- Wash the above sensor three times with PBS buffer, and then immerse it in 1% bovine serum albumin (BSA) solution to block the remaining unbound sites and then wash it with PBS buffer.

A schematic diagram of the fiber sensor surface

modification process is shown in Fig. 6(a-d). After functionalization, the fiber sensor can be used for detection of hCG. Once the hCG antigen was specifically captured by the hCG- β -mAb immobilized on the fiber surface as shown in Fig. 6(e), both effective RI and diameter of the fiber sensor increase, resulting in change of the spectral response. The concentration of hCG can be determined significantly by calibrating the wavelength shift of dips.

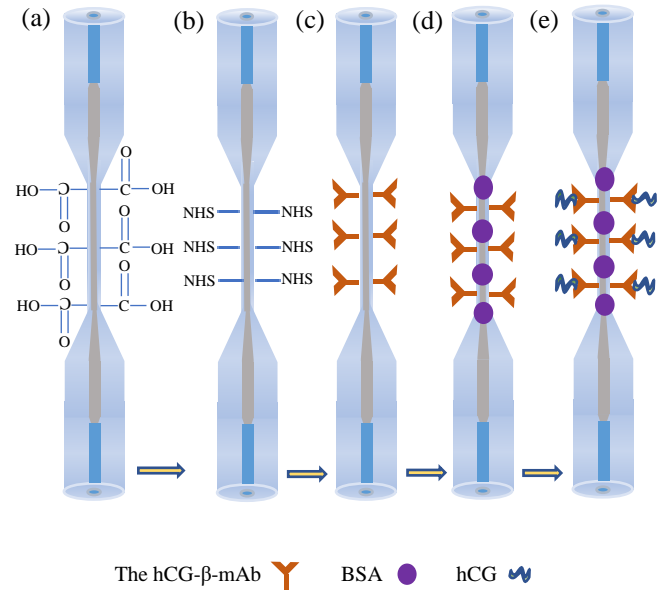


Fig. 6. Schematic diagram of fiber surface modification process: (a) treat with silane reagent to create carboxyl group; (b) generate NHS active ester with EDC/NHSS; (c) immobilize hCG- β -mAb on the fiber sensor surface; (d) block unbound sites with BSA; (e) specific binding with hCG.

Figure 7(a) shows the spectral responses of the fiber sensor (modified with 15 $\mu\text{g}/\text{ml}$ hCG- β -mAb, marked as fiber sensor 1) vs. time when it was immersed into hCG solution with concentration of 5 mIU/ml. The spectral response changed rapidly in the first 15 minutes and then stabilized at around 20 minutes, indicating specific binding between hCG- β -mAb and hCG antigen was mainly taken place at the first 15 minutes and then saturated. Figure 7(b) summarized the measured wavelength shifts of the STHS biosensor vs. time when it was immersed in different concentration of hCG solution, where a wavelength shift of 0.5, 0.8, and 1.7 nm has been observed for hCG concentration of 5, 50, and 500 mIU/ml respectively. Assuming the wavelength shift is linear when the hCG concentration is less than 5 mIU/ml, the sensitivity is thus $0.5/5=0.1 \text{ nm}/(\text{mIU}/\text{ml})$. Since the maximum wavelength variation of the sensor in phosphate buffered saline (PBS) is $\pm 0.02 \text{ nm}$ over 35 minutes, the limit of detection (LOD) of hCG can be estimated as 0.6 mIU/ml ($0.06/0.1=0.6$) assuming 3 times of the maximum wavelength variation (0.06 nm) in PBS is defined as measurement limit. Figure 7(c) shows that as hCG concentration increases, the wavelength shift increases monotonically. The reproducibility of the sensor was investigated by fabricating the identical STHS fiber structures and functionalize them with the same conditions and use them to do the same test as above. The measurement results were shown in the Fig. 7(c), which indicates relatively good reproducibility. Specificity of the sensor is investigated

by immersing the sensors into 7×10^5 CFU/ml *Staphylococcus aureus* (*S. aureus*), 10 mg/ml BSA and 4×10^6 CFU/ml *Escherichia coli* (*E. coli*) solution and the result is shown in Fig. 7(d), which indicates that the developed sensor has good specificity.

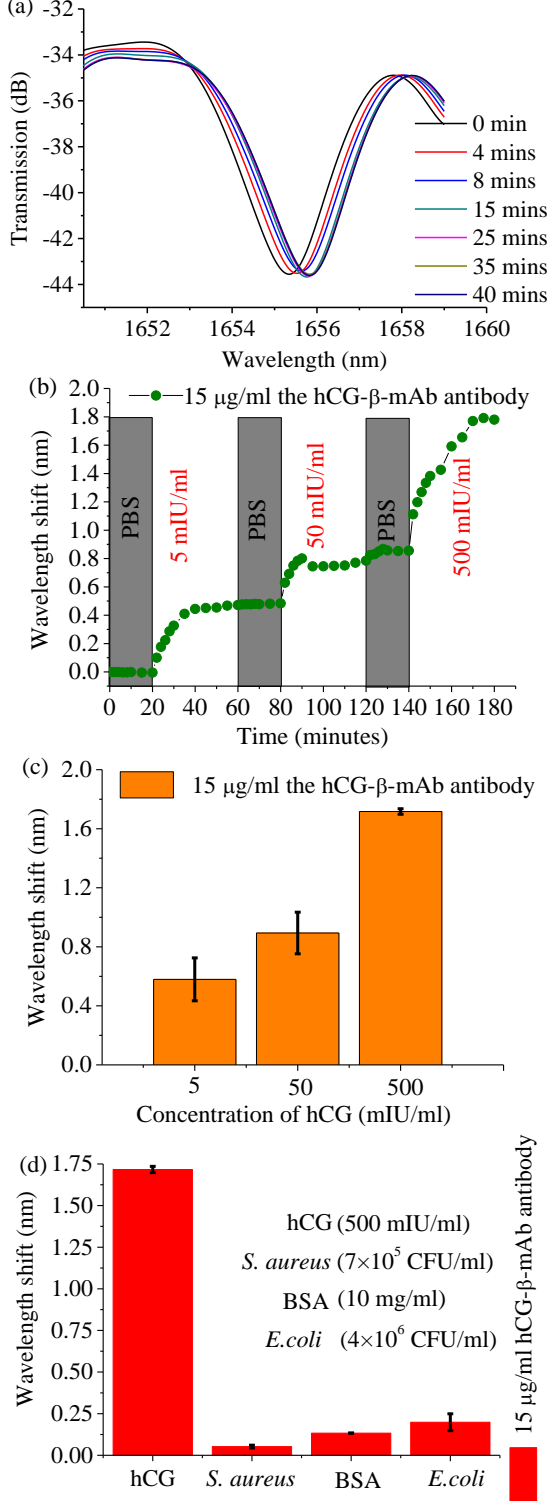


Fig. 7. Measured (a) spectral response variations of the sensor at the hCG concentration of 5 mIU/ml; (b) wavelength shift vs. time in different concentrations of hCG solution; (c) reproducibility: wavelength shift vs. concentration of hCG; (d) specificity results of the biosensor.

IV. CONCLUSIONS

In conclusion, a STHS fiber structure based on mode interference was proposed and investigated. Numerical simulations showed that the sensor structure with taper waist diameter of $26.5 \mu\text{m}$ has RI sensitivity of 885, 1517, and 4540 nm/RIU at RI ranges 1.33, 1.37, and 1.41 respectively, which verified by experimental results of 816, 1601.86, and 4775.5 nm/RIU respectively in the same RI range. **The temperature sensitivities depend on taper waist diameters**, where smaller diameter has larger temperature sensitivity and experimentally $26.82 \text{ pm}/^\circ\text{C}$ has been achieved with a taper waist diameter of $21.4 \mu\text{m}$. The structure can also be used for strain sensor, where $7.62 \text{ pm}/\mu\text{e}$ has been achieved with a taper diameter of $20.3 \mu\text{m}$. The developed STHS fiber structure is then functionalized with $15 \mu\text{g/ml}$ hCG- β -mAb for hCG detection, demonstrating its biosensing capability. The biosensor has very good stability with only $\pm 0.02 \text{ nm}$ wavelength variations in PBS over 35 minutes. Experimentally 0.5 nm wavelength shift has been observed for hCG concentration of 5 mIU/ml , which equivalent to LOD of 0.6 mIU/ml . The developed biosensor has relatively good reproducibility and specificity, demonstrating a good potential for real applications such as medical diagnostics.

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Ling Chen is a graduate student with the School of Measuring and Optical Engineering, Nanchang Hangkong University. Her research interest is fiber optic sensing.



Bin Liu received his B.S. and Ph.D. degree from Sun Yat-sen University, China. Dr. Liu is an associate Professor with Key Laboratory of Nondestructive Test (Ministry of Education) of Nanchang Hangkong University, China. His main research interest is fiber optic sensing.



Juan Liu received her Ph.D. degree from Beijing Normal University, China. She is a lecture with Key Laboratory of Nondestructive Test (Ministry of Education) of Nanchang Hangkong University, China. Her main research interest is fiber optic sensing.



Sheng-Peng Wan received the B.S. and Ph.D. degrees from University of Electronic and Technology of China. He is a professor with Key Laboratory of Nondestructive Test (Ministry of Education) of Nanchang Hangkong University, China. His main research interest is fiber optic sensing.



Tao Wu received his Ph.D. degree in Optics from University of the Littoral Opal Coast and Anhui Institute of Optics and Fine Mechanics, China. In 2010, Dr. Wu joined Key Laboratory of Nondestructive Test (Ministry of Education) of Nanchang Hangkong University, China. His main research interest has been the development of high-sensitivity laser spectrometer for laboratory and field studies of atmospheric trace gases and aerosols.



Jinhui Yuan received the Ph.D. degree in physical electronics from Beijing University of Posts and Telecommunications (BUPT), Beijing, China, in 2011. He is currently a Professor at the Department of computer and communication engineering, University of Science and Technology Beijing (USTB). He was selected as a Hong Kong Scholar at the Photonics Research Centre, Department of Electronic and Information Engineering, The Hong Kong Polytechnic University, in 2013. His current research interests include photonic crystal fibers, silicon waveguide, and optical fiber devices. He is the Senior Members of the IEEE and OSA. He has published over 200 papers in the academic journals and conferences.



Xian Zhou received the Ph.D. degree in electromagnetic field and microwave technology from Beijing University of Posts and Telecommunications (BUPT), Beijing, China, in 2011. She is currently a Professor at the Department of computer and communication engineering, University of Science and Technology Beijing (USTB). She was selected as a Hong Kong Scholar at the Photonics Research Centre, Department of Electronic and Information Engineering, The Hong Kong Polytechnic University, in 2013. Her research interests are focused on high-speed optical communications, short reach communications, and digital signal processing. She has published over 150 papers in the academic journals and conferences.



Keping Long (SM'06) received the M.S. and Ph.D. degrees from the University of Electronic Science and Technology of China, Chengdu, China, in 1995 and 1998, respectively. From September 1998 to August 2000, he was a Postdoctoral Research Fellow with the National Laboratory of Switching Technology and Telecommunication Networks, Beijing University of Posts and Telecommunications (BUPT), Beijing, China. From September 2000 to June 2001, he was an Associate Professor with BUPT. From July 2001 to November 2002, he was a

Research Fellow with the ARC Special Research Centre for Ultra Broadband Information Networks, University of Melbourne, Melbourne, Australia. He is currently a Professor and Dean with the School of Computer and Communication Engineering, University of Science and Technology Beijing. He has authored more than 200 papers and has delivered 20 keynote speeches. He is a Member of the Editorial Committee of Sciences in China Series F and China Communications. His research interests are optical internet technology, new-generation network technology, wireless information networks, value-added service, and secure technology of networks. Dr. Long has been a Technical Program Committee (TPC) and International Steering Committee member for COIN2003/04/05/06/07/08/09/10, IEEE IWCN2010, ICON04/06, and APOC2004/06/08; a Co-chair of the organization membership for the 2006 International Wireless Communications and Mobile Computing Conference; the TPC chair of COIN2005/2008; and a TPC Cochair of COIN2008/2010. He has been invited to talk at both international and local conferences. He received the National Science Fund for Distinguished Young Scholars of China in 2007 and was selected as the Chang Jiang Scholars Program Professor of China in 2008.



Liyang Shao received his Ph.D. in Optical Engineering in Zhejiang University in 2008. He worked in many world leading universities and research institutions, like Carleton University, The University of Sydney, The Hong Kong Polytechnic University, Nanyang Technological University. He is currently an Associate Professor at Southern University of Science and Technology of China. His main research areas are distributed optic fiber, fiber grating, fiber laser, photonic crystal fiber devices, micro-Nano fiber devices and sensing, microwave photon sensing measurement, etc.



Yong Qing Fu was a Reader in Thin Film Centre in University of West of Scotland, Glasgow, UK, and a lecturer in Heriot-Watt University, Edinburgh, UK. He obtained his PhD degree from Nanyang Technological University, Singapore, and then worked as a Research Fellow in Singapore-Massachusetts Institute of Technology Alliance, and a Research Associate in University of Cambridge. He has extensive experience in smart thin film/materials, biomedical microdevices, energy materials, lab-on-chip, micromechanics, MEMS, nanotechnology, sensors and microfluidics. He has established a worldwide reputation from his pioneer research work on shape memory films, piezoelectric thin films, nanostructured composite/films for applications in MEMS, sensing and renewable energy applications. He is currently a Professor at Northumbria University, UK.



Xing-Dao He was born in Jingan, China, in 1963. He received the Ph.D. degree in optics from Beijing Normal University, Beijing, China, in 2005. He is currently a Professor with the Key Laboratory of Nondestructive Test (Ministry of Education), Nanchang Hangkong University,

China. His current research interests include light scattering spectroscopy, optical holography, and information processing.



Qiang Wu received the B.S. and Ph.D. degrees from Beijing Normal University and Beijing University of Posts and Telecommunications, Beijing, China, in 1996 and 2004, respectively. From 2004 to 2006, he worked as a Senior Research Associate in City University of Hong Kong. From 2006 to 2008, he took up a research associate post in Heriot-Watt University, Edinburgh, U.K. From 2008 to 2014, he worked as a Stokes Lecturer at Photonics Research Centre, Dublin Institute of Technology, Ireland. He is an Associate Professor / Reader with Faculty of Engineering and Environment, Northumbria University, Newcastle Upon Tyne, United Kingdom. His research interests include optical fiber interferometers for novel fiber optical couplers and sensors, nanofiber, microsphere sensors for bio-chemical sensing, the design and fabrication of fiber Bragg grating devices and their applications for sensing, nonlinear fibre optics, surface plasmon resonant and surface acoustic wave sensors. He has over 200 publications in the area of photonics and holds 3 invention patents. He is an Editorial Board Member of Scientific Reports, an Associate Editor for IEEE Sensors Journal and an Academic Editor for Journal of Sensors.